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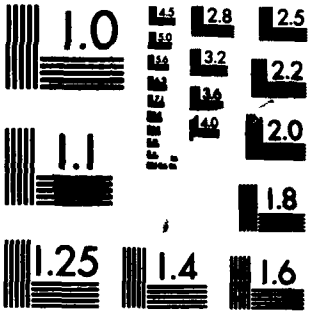

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Ion implantation and related ion-beam processing techniques are used to modify the surface of materials and produce certain desirable properties. However, these methods sometimes roughen the surfaces to which they are applied. If undetected, such roughness can lead to erroneous interpretation of data gathered by most standard surface analysis techniques. Many surface profilometers and scanning electron microscopes lack sufficient spatial resolution to detect fine-scale roughness that can complicate the

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"OPTICAL MEASUREMENT OF THE RMS ROUGHNESS OF ION-BOMBARDED SURFACES"

A Trident Scholar Report

by

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Accepted for Trident Scholar Committee

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Abstract

Ion implantation and related ion-beam processing techniques are used to modify the surface of materials and produce certain desirable properties. However, these methods sometimes roughen the surfaces to which they are applied. If undetected, such roughness can lead to erroneous interpretation of data gathered by most standard surface analysis techniques. Many surface profilometers and scanning electron microscopes lack sufficient spatial resolution to detect fine-scale roughness that can complicate the data interpretation. A simple optical instrument has been constructed to measure the root-mean-square (rms) roughness, below about 100 nm, of ion bombarded surfaces. This instrument measures the total integrated scatter (TIS) of almost normally incident laser light, which (under conditions specified by scalar scattering theory) is simply related to the rms surface roughness. This paper describes the construction and calibration of the TIS instrument. In addition, it presents results on the rms roughness of several ion-beam-processed systems, including TiN films on Si, Cr and Cr_2O_3 films on AISI 52100 steel, ion-beam-mixed Mo in Al, Si_xN_{1-x} refractive layers, and GaAs/AlAs superlattices.

Acknowledgements

Many people have provided help along the road from inception to final completion of the project. I would like to mention as many of those people as possible who devoted their time, resources, and experience to my research.

The optical instrument could never have been built without the metal-working and machine shop skills of Mr. Charlie Holloway, the loan of needed optical equipment from Associate Professor Lawrence L. Tankersley, and the use of Professor John J. Fontanella's vacuum deposition apparatus. Mr. John M. Guerra, P. E., of Polaroid Optical Engineering provided essential details concerning his TIS instrument. Many helpful telephone conversations with Dr. Jean M. Bennett, an expert in the field of surface analysis, are greatly appreciated. Dr. Joseph M. Argento is thanked for arranging the loan of the roughness standards. Most of the samples measured, and much important information about ion-beam processing, were obtained from the U. S. Naval Research Laboratory; in particular, the help of Dr. Richard A. Kant and Dr. Graham K. Hubler are gratefully recognized. I will never forget or stop being thankful for the time and effort that my advisor, Associate Professor F. D. Correll, dedicated to the project. Finally, none of this research would have been possible without the funding and encouragement of the Naval Academy Research Council and the Trident Scholar Committee.

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1. Introduction

"Our world is a material world. Everything we see, touch, taste, or smell is composed of one or more materials. ... Interest in the matter that makes up our environment has grown with the realization that it can be controlled."[1]

These words concisely express the purpose of materials science, the general subject area of this report. Materials science can be also described (perhaps somewhat more narrowly) as the study of methods to characterize and improve the properties of materials. In an increasingly technological society, the need for materials with special properties becomes increasingly great. New metals are needed to make high-speed bearings that will not corrode in hostile environments. New semiconductors are needed to make ever-faster, higher-density integrated circuits that can function at high temperatures or in high radiation fields. New kinds of superconductors are needed to make power transmission lines or high-field magnets that will operate at near room temperature. Many researchers are actively engaged in basic and applied research that contributes to these goals.

Many different techniques have been developed to improve the raw materials nature provides. One very promising technique developed over the last twenty or thirty years may be called "ion bombardment." Although this term can be applied to several processes that differ in detail, the basic method is common to all: high speed ions of selected chemical elements are violently driven into the near surface region of

the materials to be modified. As described in section 2 of this report, this general approach has been used to electrically dope semiconductors; to improve the friction, wear, and corrosion properties of many different metals; and to produce thick silicon-nitride layers with smoothly varying indices of refraction which act as sturdy, highly selective filters for infrared radiation.

The many benefits of ion bombardment do not come without side effects. One important side effect is that ion bombardment sometimes roughens the surfaces to which it is applied. A being the size of an atom on the surface of such a roughened object would experience a new, exotic world filled with canyons, steep cliff faces, and mountains. These rough surfaces may be either desirable or undesirable, depending upon the object's intended use. In either case, if roughness is present, the materials scientist usually wants to know about it.

Two traditional methods of investigating surface roughness are mechanical profilometry and electron microscopy, both of which can be time consuming and relatively difficult to apply. In addition, the resolution needed to detect fine-scale roughness that can affect data interpretation may be lacking. A rapid, simple, reasonably quantitative method of assessing surface roughness would be beneficial.

A promising method, known as total integrated scatter (TIS), has been used for many years to measure the microroughness of precision optical components.[2] As described in section 4, the TIS technique is based on the fact that a rough surface reflects light more diffusely than a smooth one. Under conditions that apply to many ion-bombarded surfaces, the intensity ratio of diffusely scattered light to all

reflected light can be related in a straightforward way to the root-mean-square (rms) roughness of the reflecting surface.

As the primary objective of this Trident Scholar project, a simple, inexpensive optical instrument has been constructed at the U. S. Naval Academy to measure the rms roughness of ion-implanted surfaces. The TIS instrument has been used to study several different ion-bombarded materials, particularly thin Cr and TiN films produced at the U. S. Naval Research Laboratory (NRL). The construction and calibration of the instrument are described in section 5; descriptions of the samples and results of the roughness measurements are found in sections 6 through 10. A brief summary, contained in section 11, concludes this report.

2. Material Modification

The surface of an object is a dynamic interface, where atoms of the material comprising the object meet and interact with their surroundings. Many important characteristics of an object (or the material of which it is made), such as its resistance to wear and corrosion, its coefficient of friction, and its optical reflectivity, are determined by the physical, chemical, and structural properties of its surface.

For centuries, many different techniques have been devised to tailor the properties of materials by modifying their surfaces. Familiar, traditional techniques include painting, lubricating, plating, and various kinds of heat treating. Although these approaches are generally quite successful, they have some disadvantages. One common problem is that surface coatings, like paint and plated layers, frequently do not adhere well, especially if they become damaged and the underlying surface becomes exposed. Another problem is that some processes, like heat treating, may adversely affect the bulk properties of the material while concurrently improving its surface properties. In the past few years, new methods have been developed which circumvent these difficulties and make it possible to create durable surfaces with desirable properties on many different materials.

2.1 Material Modification by Ion Implantation

Ion implantation is one especially promising new technique in which atoms of selected chemical elements are ionized, focussed, accelerated through a high potential difference, and directed at high speed onto the surface to be modified. A specialized particle accelerator called an "ion implanter" is used to carry out the process. A typical ion implanter is shown in Figure 1; such an implanter usually produces ion beams with kinetic energies in the range from 10 to 500 keV. At these energies, the ions may penetrate from 10 to 1000 nm into the target material, depending on their mass and the properties of the material itself.[3]

The implanted atoms modify the properties of the near-surface region through various mechanisms: for example, by changing its chemical composition, creating atomic displacements, and introducing compressive stress.[4] Since ion implantation is a violent, non-equilibrium process, the resulting changes in composition and structure are not governed by solid solubilities, diffusion rates, or other thermodynamic constraints that limit most conventional techniques. As a result, ion implantation can be used to endow surfaces with novel chemical, electrical, mechanical, or optical properties without affecting the bulk characteristics of the material.

Until recently, the most extensive application of ion implantation has been in the semiconductor industry. Beginning in the 1970's, developments in ion implantation and in furnace annealing were combined

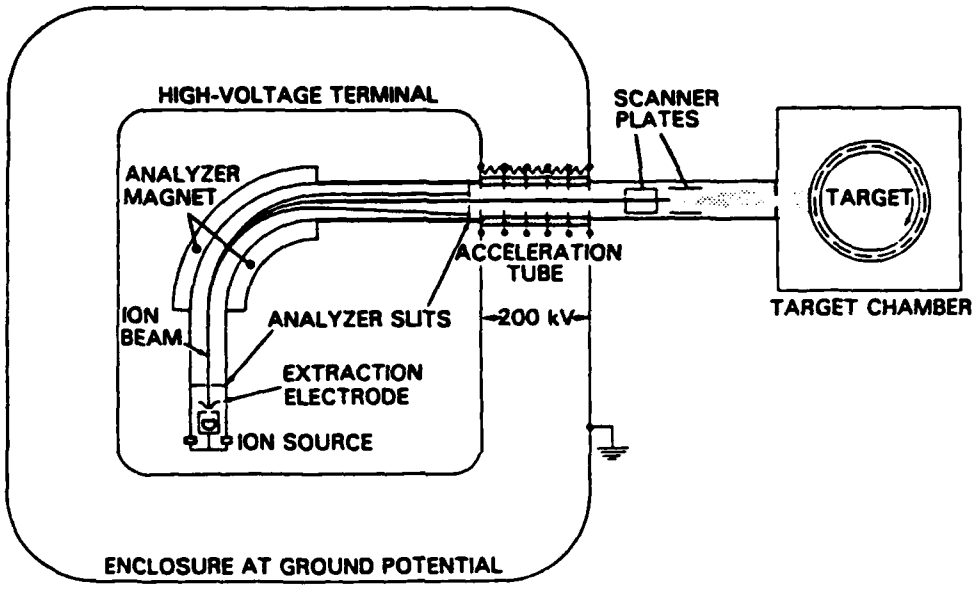

Figure 1. Schematic diagram of an ion implanter used to modify materials by ion bombardment. Atoms of the chosen element are ionized, focussed by electric and magnetic fields, mass selected, and finally accelerated through a high potential difference before striking the target material. To insure uniform coverage, the accelerated ion beam is scanned across the target in a manner similar to that of a TV raster.

to produce a new, commercially acceptable method of electrically doping silicon, which has now largely replaced doping by the traditional method of diffusion.[5] This new process was especially attractive because of its reproducibility and its compatibility with other steps in the processing chain. In particular, ion implantation introduces dopant atoms in a dry, athermal process using a beam that can be focussed to small dimensions. As a result, small, electrically active elements can literally be "drawn" into the substrate of an integrated circuit chip by ion implantation, and the temperature of the substrate can be adjusted independently to provide annealing of defects or control the mobility of the implanted species.

In the last decade, ion implantation has been applied to a wide range of other materials and systems. For example, ceramics have been implanted to increase their toughness[6]; polymers have been implanted to stimulate ion or electron conductivity[7]; and dielectric materials have been implanted to form light guides.[2]

One rapidly growing field, with great potential for economic impact, is ion implantation of metals and alloys. A large body of research performed during the last few years has demonstrated that many different metals can be made more resistant to wear and corrosion by implanting them with selected ions. For example, the useful lifetime of certain expensive steel metal-forming tools (like stamps and dies) or injection molds (for plastic casting) can be increased by factors of 2 to 10 by implanting them with energetic nitrogen (N) ions.[8] Nitrogen implantation can also improve the properties of surgical titanium (Ti) alloys, which are used to make prostheses like artificial hip joints.[9]

A typical artificial hip joint might consist of a Ti-alloy ball that rotates in a polyethylene socket; in the saline environment of the body, corrosive wear of the ball joint may occur, producing debris that can inflame the surrounding tissues. Implantation with N ions has been shown to dramatically decrease the rate of corrosive wear of these alloys; experimental results imply that the lifetime of artificial joints may be extended by factors of as much as 400. As a final example, joint implantation of both Ti and carbon (C) into soft (type 304) stainless steel has been shown to produce a hard, amorphous surface alloy with a coefficient of friction about half that of unimplanted material, and a wear rate two to five times smaller than that of unimplanted material.[10,11]

2.2 Roughening of Surfaces During Ion Bombardment

When energetic ions strike the surface of a material, they typically deposit their kinetic energy within a few atomic layers. This energy is rapidly distributed throughout the near surface region by collisions. Sometimes an individual target atom receives enough energy to be ejected from the surface of the material, in a process called sputtering.

If the surface of an object sputtered uniformly, the surface would simply recede, and no topographical changes would occur. However, this is not the case.[12] The sputtering probability or "yield," depends upon the mass of the projectile atom, the mass of the target atom, and the surface binding energy of the target atom. In addition, the

crystallographic orientation of the grains that make up the material, and the macroscopic orientation of the material's surface, influence the sputtering yield.

Because the sputtering yield is not the same for all parts of a surface, sputtering can modify the topography of the surface in a number of ways. Crystal grains with certain crystallographic orientations can be preferentially sputtered. Some target atoms may be especially weakly bound to the surface, allowing them to be preferentially removed. Pre-existing inhomogeneities, like pits, steps, or steeply sloped regions, can be enhanced by ion bombardment. The result of all of these processes is the roughening of ion-bombarded surfaces.

3. Mathematical Description of Rough Surfaces

Producing a complete, accurate description of a rough surface is an enormously complex task. In principle, such a description would consist of a very large collection of numbers representing the height of the surface at a very large number of chosen positions. Data of this kind might be obtained, for example, using a stylus profilometer; many closely-spaced, one-dimensional traces of the surface could be made and combined to yield a kind of microscopic topographical map.

Fortunately, such detail is not required for many studies of surface roughness. This seems to be true of the surfaces produced by ion bombardment, which are often described as "randomly rough"; that is, they have neither a preferred orientation nor a preferred spacing. An example of a surface that is not random is a diffraction grating, which consists of many long, parallel grooves, each accurately separated from its neighbors by the same distance. On the other hand, a randomly rough surface might be compared to a collection of superimposed diffraction gratings with randomly selected ruling spacings and orientations.

For such surfaces, it is often sufficient to specify the root-mean-square (rms) roughness δ (which describes the "amplitude" of surface features) and the autocovariance length T (which describes the "spatial wavelength" of the surface).[13] Both of these measures can be derived mathematically from a height distribution w(z), which for a randomly rough surface is well approximated by a Gaussian distribution:

$$w(z) = (1/\delta\sqrt{2\pi})\exp[-(z/\delta\sqrt{2})^2]. \qquad (1)$$

The rms roughness represents the rms deviation from the mean surface, which is the perfect surface that would be formed if all the roughness peaks were cut off and used in just filling up the valleys below that surface. Mathematically, the rms roughness can be calculated from the expression

$$\delta = [(1/n)\sum z^2(i)]^{1/2}, \qquad (2)$$

where the height of the surface, relative to its mean height, is measured at n points, and z(i) represents the height of the ith point.

Different measuring techniques are sensitive to different ranges of spatial wavelengths. For this reason, it is often difficult to compare roughness measurements made using different techniques.

4. Theory of Total Integrated Scatter

Scalar scattering theory is the framework used to analyze the surface roughness data collected by most optical techniques. Certain assumptions have been made to make data interpretation relatively simple, manageable, and meaningful. A surface being measured is considered to be "slightly rough" if its rms roughness (δ) is much smaller than the wavelength (λ) of the light illuminating it.

A quantity called total integrated scatter (TIS) is defined as the intensity ratio of diffusely scattered light to all reflected light. Because a rough surface reflects light more diffusely than a smooth one does, the TIS increases with roughness. For a perfectly smooth surface, TIS equals zero, while the TIS for a "perfect diffuser" is equal to unity.

When the surface is only slightly rough, the corresponding TIS can be computed using scalar scattering theory; the result[14] is:

$$TIS = 1 - \exp[-(4\pi\delta\cos\theta/\lambda)^2], \qquad (3)$$

where θ is the angle of incidence. If the TIS from a surface is measured, then its rms roughness can be calculated.

Unless one is careful to collect light scattered at very small angles from the specular direction, the TIS technique is sensitive to only short-wavelength components of the roughness.[15] Therefore, difficulties sometimes arise when comparing TIS results with those of different measurement techniques like mechanical profilometry.

5. USNA TIS Instrument

The USNA Total Integrated Scatter instrument was based on a design developed by John M. Guerra, P. E. of Polaroid Corporation[16]. The principal components of the instrument are shown in Figure 2; details of the construction are shown in Figures 3 and 4.

5.1 Design and Operation

Red light of wavelength $\lambda = 632.8$ nm is produced by a 5 mW HeNe laser (Spectra-Physics model 105-2) and enters an enclosed box, which is painted black to exclude room light, through a hole approximately 5 mm in diameter. A prism directs the laser light at a near normal angle through a 2 mm diameter hole close to the apex of an aluminized acrylic hemisphere. After traversing a 5 mm hole in a mounting plate at the base of the hemisphere, the light strikes the sample which is offset slightly from the center of the hemisphere. The sample rests on a 7.62 cm × 7.62 cm aluminum plate with three adjustable cap screws that are used to tilt it slightly for optimum angular alignment.

If the sample is rough, it will scatter the laser light into all angles of the hemisphere. This scattered, or diffusely reflected, light is collected by the hemisphere and is focussed onto a point that is offset from the center by the same amount as the sample, but in the opposite direction. A silicon photovoltaic cell (Hamamatsu model S1337-1010BR) fits into a rectangular hole at this conjugate focal point. The

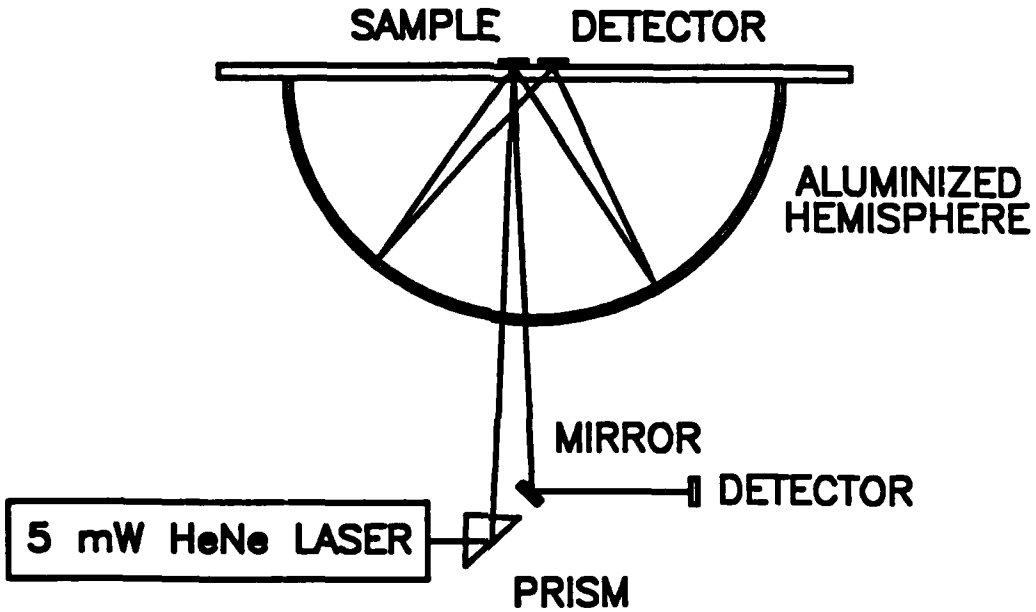

Figure 2. Principal components of the USNA Total Integrated Scatter instrument. The alignment laser mentioned in the text is not shown, nor are the mechanical supports for the various components.

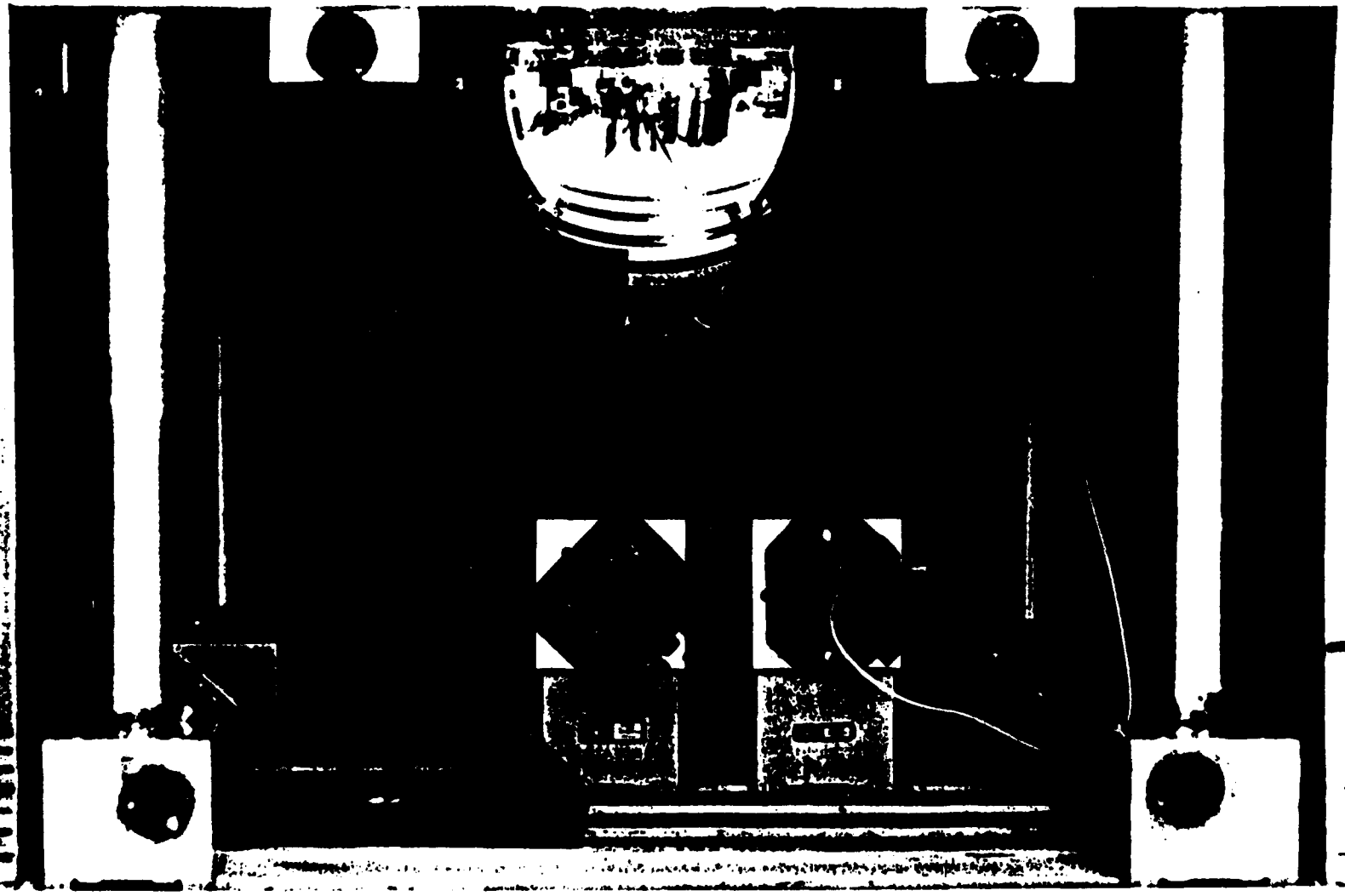

Figure 3. Photograph of the TIS instrument, showing details of the mechanical construction. This view is through the front panel of a light-tight box that is normally closed during use. The sample rests on the top plate near the upper center of the photograph, and light from the 5-mW laser enters from the lower left. Just visible at the upper right is a small box containing a reed relay which switches the input of digital multimeter from one photocell to the other under control of the instrument's computer.

Figure 4. Photograph of the TIS instrument, with the room lights extinguished and the box filled with cigarette smoke to make the laser beam visible. The sample used in this photograph was a smooth mirror, which produced an intense specularly-reflected beam. This beam leaves the hemispherical mirror through a small hole just to the right of center in this view. After being deflected by a small diagonal mirror, it strikes the photocell and is reflected onto a piece of black velvet which acts as a beam dump. In a later version of the instrument, this reflected beam is directed instead onto a ground-glass screen to aid in aligning the sample.

light that has not been scattered, called specularly reflected light, exits an approximately 2 mm diameter hole near the apex of the hemisphere, which is about 1 mm from the entrance hole. The specularly reflected beam bounces from a mirror onto another photocell, identical to the first. A ground glass reference screen, used to determine proper alignment, intercepts the light reflected from this photocell.

Currents from the two photocells are converted to voltages by being dropped across a 10-Ω resistor; these voltages are then directly proportional to the specularly and diffusely reflected intensities. The voltages are measured using a digital multimeter (Keithley model 197) which is alternately connected to one photocell or the other by a computer-controlled reed relay. The measured intensity readings are sent via an IEEE 488 interface to an Apple IIe personal computer which averages five readings of each intensity to reduce statistical fluctuations. A schematic diagram of the principal electrical components is shown in Figure 5. Using a program based upon scalar scattering theory (see Appendix 2), the Apple IIe analyzes the data received. The program calculates the total integrated scatter and root mean square roughness of the sample spot observed. An Okidata μ92 printer prints out these values. After the operator determines that sufficient data have been taken, he can have the computer program stop recording data and print out averages and standard deviations of the results.

Before any roughness measurements can be made, the light beam must be properly aligned. A ground glass reference screen helps to accomplish this task. The placement of the reference screen is done by measuring the roughness of a smooth piece of Si wafer (Monsanto lot #1105D). An

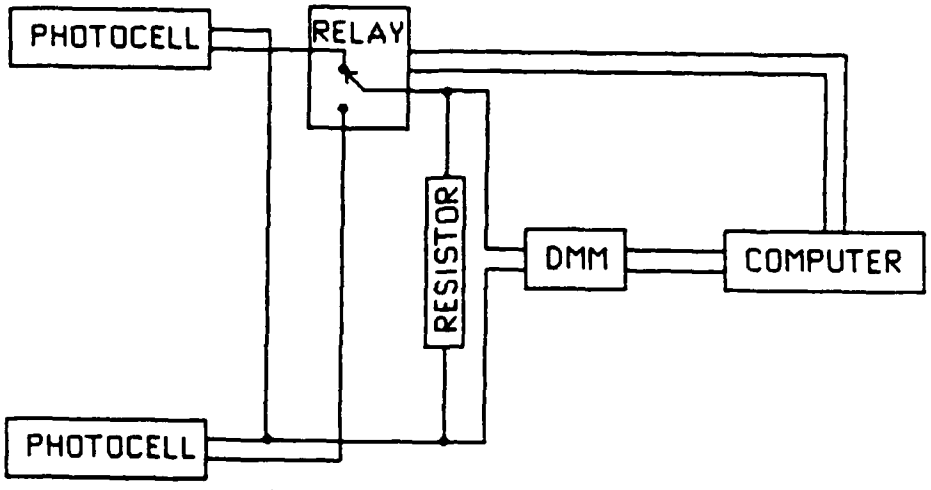

Figure 5. Principal electronic components of the TIS instrument. Each of the silicon photovoltaic cells may be selected by a reed relay, and its output current measured by a digital multimeter (DMM) connected to a small computer. The DMM communicates with the computer via an IEEE-488 interface; the computer drives the relay through a game port connector.

Applesoft Basic program, written for the Apple IIe computer (see Appendix 1), is used to compute the rms roughness of the Si wafer while the operator adjusts the cap screws on the sample holder plate. The screws are individually turned until the computer displayed the smallest reading possible. Once the operator to his satisfaction has reached the smoothest value practical for the rms roughness, crosshairs are drawn on the ground glass screen to mark the laser spot. The crosshairs indicate the locus of optimum alignment for the sample being measured.

Locating the exact sample spot the laser contacts is essential, for many samples have scratches and pits produced after processing that must be avoided for a relevant measurement. A Melles-Griot 0.5 mW red light laser is directed by a mirror at 45 degrees to the incident beam onto the "back" side of the sample. A rod and clamp arrangement holds the mirror securely. Both laser beams travel perpendicular to the operator's line of sight, to preclude any eye damage from the laser light. The spotting laser can be adjusted to intersect the measuring light beam at the sample position. The intersection of the two beams varies insignificantly over the course of a day's measurement of materials.

The total cost of the TIS instrument, except for the computer and a small optical table, was about $1,600.

5.2 Instrument Calibration

The TIS instrument was calibrated using a set of fifteen roughness standards developed at the Naval Weapons Center (NWC), China Lake, CA, for the U. S. Army. The standards consisted of polished and aluminized

glass or quartz disks. Their rms roughnesses were measured by TIS at NWC after fabrication, and again after they had been used at several laboratories. The original δ ranged from 0.42 to 33 nm; the later ones ranged from 0.74 to 39 nm. The later NWC δ and those measured using the USNA TIS instrument are plotted versus the original δ in Figure 6.

Except for the smoothest standards, the USNA and NWC measurements generally agree within ±15% or ±1.5 nm, which may be considered to be an estimate of the absolute accuracy of the USNA TIS instrument. For the smoothest standards, the lowest rms roughness value obtained with the USNA instrument is about 2 nm. This can probably be attributed to low-level light scattering in that instrument, and suggests that it is probably not accurate for measurements below about 2 nm. Experience with a similar instrument at Polaroid Optical Engineering suggests that the USNA instrument should be accurate for roughnesses as large as 100 nm.

As a further check on the instrument's performance, the roughness of a Marz-grade Co disk polished with 9-μm alumina was measured using a mechanical profilometer and the TIS instrument. The output from the profilometer was digitized (see Figure 7), and the rms roughness of the surface was found to be approximately 27 nm. The TIS instrument yielded a value of 28±3 nm. Although roughness values obtained by TIS and by mechanical profilometry are not necessarily expected to be consistent, this agreement is encouraging.

Several tests were made to determine the reproducibility of the instrument. In these tests, the roughness of a single spot on a sample was measured several times. Figure 8 shows a graph of some typical results for a smooth Si wafer. The conclusion drawn from these tests is

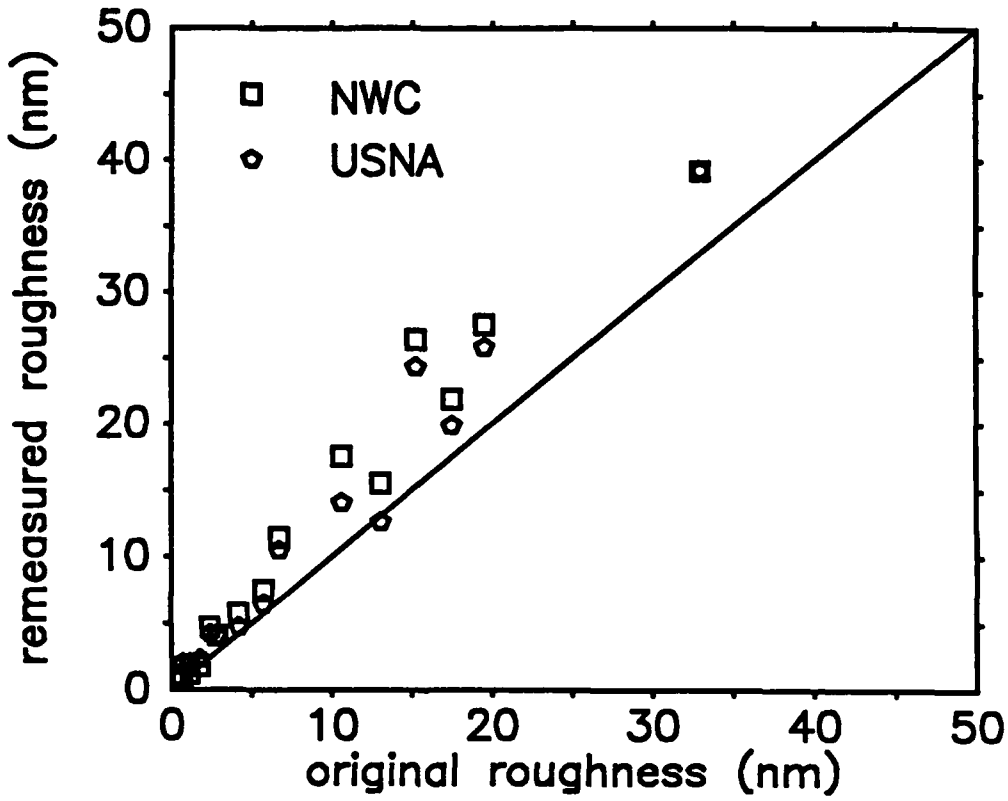

Figure 6. Performance of the USNA TIS instrument compared to that of a state-of-the-art instrument at the Naval Weapons Center, China Lake. USNA and NWC values for the roughnesses of 15 standard samples are plotted versus the roughnesses originally measured at NWC, as discussed in the text. The diagonal line indicates equal original and remeasured roughnesses.

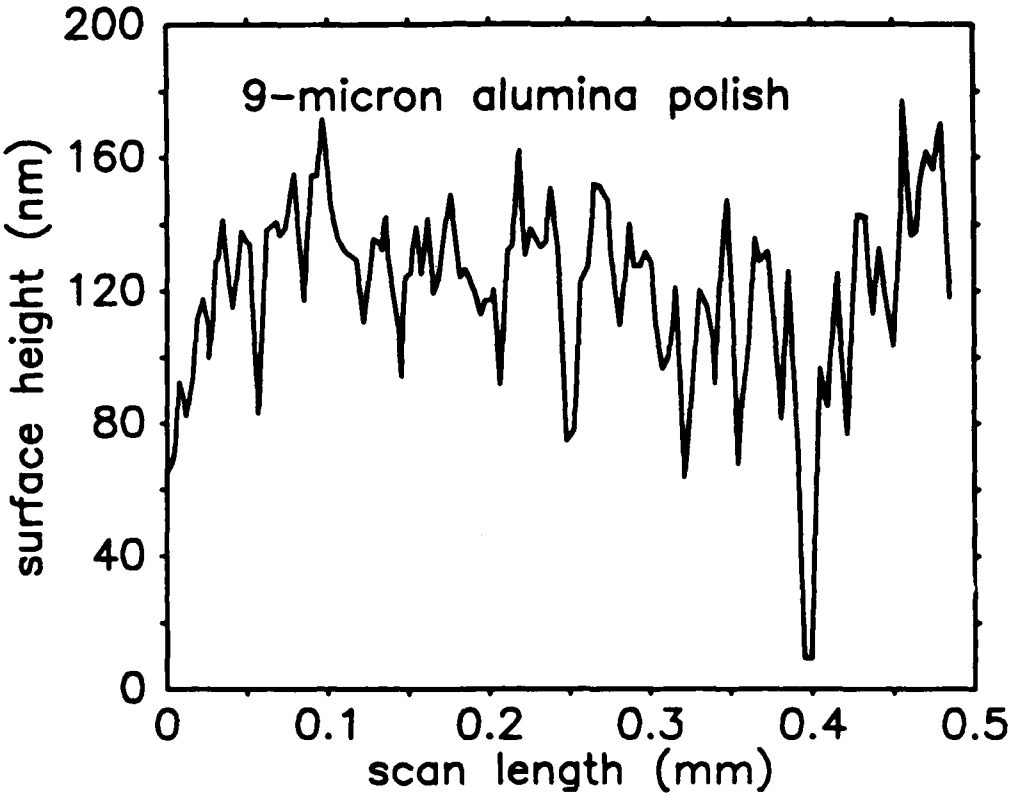

Figure 7. Surface height profile of a Co sample polished with 9-μm alumina. This profile was obtained using a Dektak stylus profilometer and was digitized using a Tektronix digitizing tablet. The rms roughness deduced from this profile was approximately 27 nm; the roughness measured with the TIS instrument was 28 ± 3 nm.

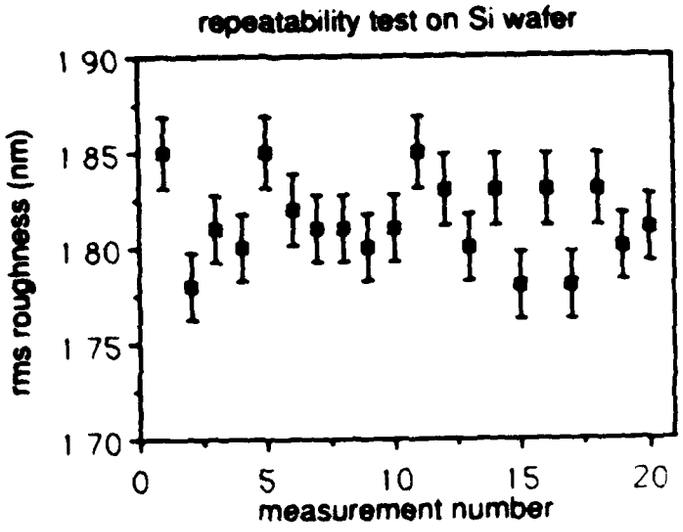

Figure 8. Results of a repeatability test of the TIS instrument. The roughness of single spot on a silicon wafer was measured twenty times, and the individual values are plotted versus measurement number. The error bars represent the change in measured roughness produced by a one-count change in the least significant digit of the digital multimeter.

that the instrument is satisfactorily reproducible, since the standard deviation of the mean of a set of twenty measurements was usually less than ±0.01 nm.

6. Application 1: TiN Films on Silicon

In recent years, a large body of research into thin film implantation has been carried out by material scientists in the Materials Modification and Analysis branch at the Naval Research Laboratory (NRL). One area of current research involves the use of ion-assisted deposition (IAD) to design coatings with low coefficients of friction and high resistance to wear and to corrosion. Ion bombardment of Si substrates with Ti or N while simultaneously evaporating Ti ions onto the surface has been found to produce a layer that is tough and adherent but is also sometimes roughened. Using the TIS instrument, extensive measurements of the rms roughness were performed on TiN samples procurred from Richard Kant and other researchers at NRL.

TiN films are useful since they are highly adhesive, experience low friction and wear, and are very decorative. NRL has applied these films to increase the hardness and wear of steel cutting tools such as twist drills and lathe tools. TiN layers can act as diffusion barriers for integrated circuit chips to prevent the diffusion of aluminum into silicon.[15] A more common use of TiN films is for coating jewelry to obtain gold-like luster.

6.1 Sample Preparation

The TiN layers were deposited onto 1 cm × 1 cm smooth Si substrates that had been pre-implanted with an incident flux of $10^{16}/cm^2$ 40 keV Ti+

ions to sputter clean the surface and to create a graded Ti-Si interface for better adhesion of the deposited films. The deposition chamber was backfilled with nitrogen gas at 13 mPa, and contamination from the rest of the system was avoided by differential pumping, cryogenic pumping, and surrounding the sample with a cold wall kept at the temperature of liquid nitrogen.[18]

As Figure 9 shows, the sample made a 45 degree angle with the titanium vapor being deposited. The titanium atoms reacted with the nitrogen gas in the sample chamber to form TiN. Perpendicular to the vapor stream, 40 keV Ti^+ ions were implanted into the near surface region. Two regions of interest were produced by masking part of the sample from the ion beam but not from the vapor stream. A computer-controlled e-gun allowed Ti atoms to strike the sample at a deposition rate between 0.12-0.38 nm/s[18]; the deposited layer consisted of a 500 nm thick coating, called the physical vapor deposition (PVD) region. Covering only half of the sample at an energy of 40-keV, Ti^+ ions at a current density between 2.5-17 $\mu A/cm^2$[18] were buried into the substrate to form the ion-assisted deposition (IAD) region. However, the Ti^+ ions from the beam travelled fast enough to not appreciably react with the nitrogen in the chamber. The Ti^+ ions helped to make the deposited TiN layer adhere better to the silicon substrate.

The researchers at NRL varied the arrival rate ratio and the substrate temperature in order to investigate the effects of these variables on the properties of the TiN layers produced. The arrival rate ratio is defined as the number of bombarding ions divided by the number of vapor deposited atoms on the IAD region. Maintaining the desired

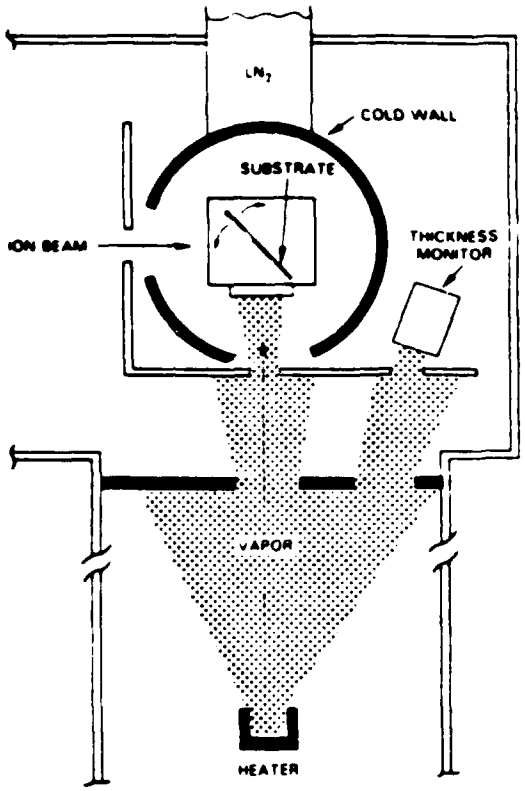

Figure 9. Schematic view of the apparatus used for ion-assisted deposition at the Naval Research Laboratory. The TiN and Cr_2O_3 films measured as part of this project were made using this apparatus. Ti or Cr vapor from the heated source at the bottom of the figure is deposited on the temperature-controlled sample at the center of the vacuum chamber. At the same time, Ti or Cr ions from an ion implanter strike the sample at right angles to the vapor stream. A thickness monitor measures the arrival rate of the vapor while a Faraday cup (not shown) and a current integrator measure the arrival rate of the ions; a computer controls the source heater input power to vary the arrival rate ratio.

arrival rate ratios required varying both the deposition rate of the incoming vapor and the current density of the ion beam. The samples studied in this project had arrival rate ratios of 0.01, 0.05, and 0.15, and they were deposited at substrate temperatures of 100, 175, 350, 525, and 700 degrees C.

6.2 Results of TIS Measurements

The measured rms roughness of the TiN films is plotted versus the arrival rate ratio R in Figure 10. Data for the TiN samples are included in Table I, and an output data sheet from a typical sample measurement is shown in Appendix 3. These results were presented in a talk on March 20, 1987, at a meeting of the American Physical Society in New York City. An abstract of that talk appears in Appendix 4.

At each temperature, the rms roughness of the PVD regions is nearly independent of R. This is a useful check of our apparatus, since the rms roughness of the PVD region is not expected to vary significantly with the arrival rate because no bombarding ions reach that region. For the low arrival rate data (except at 525°C), the IAD regions are smoother than the PVD regions. At low arrival rates, the ratio of ions implanted to TiN deposited probably can cause enough mobility of the TiN layer to spread out the film evenly. For the highest arrival rate and all of the 525°C data, IAD regions are rougher than PVD regions. If the arrival rate is too great, then the sputtering effect of the Ti^+ ion beam possibly dominates the smoothening due to the increased mobility of the TiN layer, and uneven erosion of the surface results.

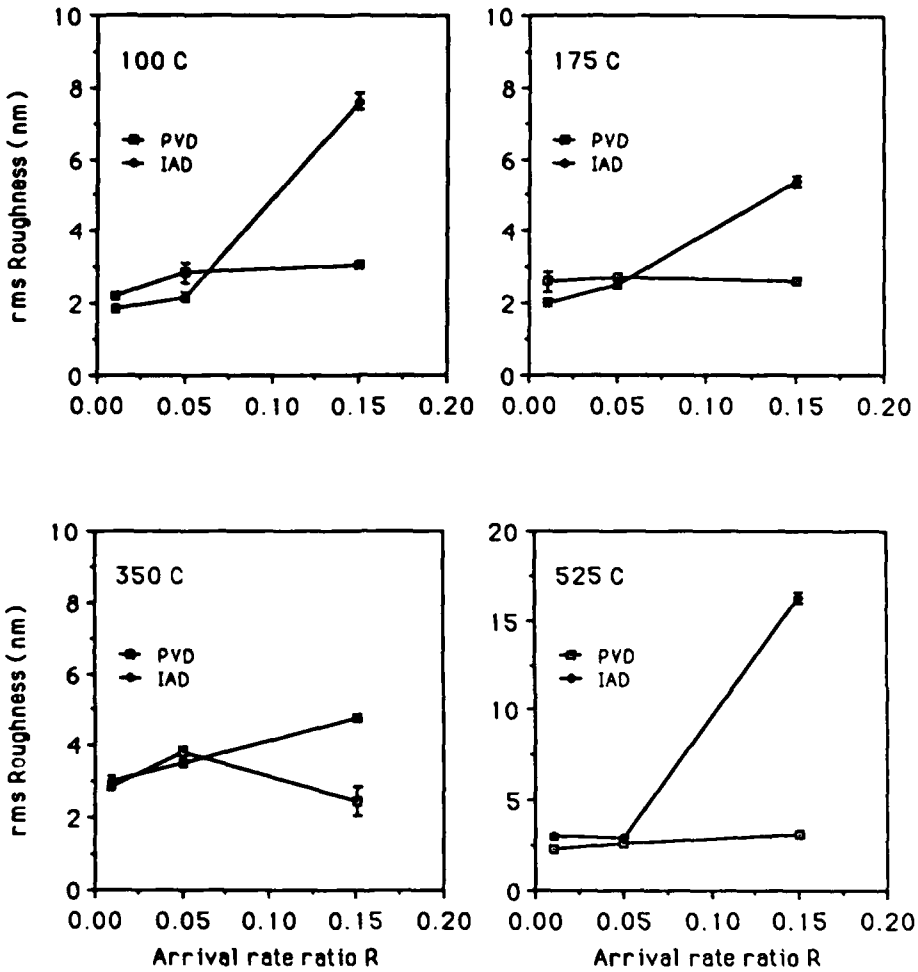

Figure 10. RMS roughness of TiN films on Si substrates. Each graph shows the results of TIS measurements on samples produced at one temperature (which is listed in the upper left-hand corner of the graph). Each sample was divided into two parts: the PVD region (square symbols), which received only a TiN vapor deposition, and the IAD region (circles), which was also bombarded by energetic Ti ions. The rms roughness of the surface is plotted against the arrival rate ratio R defined in the text.

Table I. RMS roughness of TiN films on Si. These data are also plotted in Figure 10.

Sample ID	Temp (°C)	R	Area	Thickness (nm)	Roughness (nm)
102.2.6	100	0.01	PVD	330	2.20±0.08
925.2.6	100	0.05	PVD	500	2.84±0.28
930.1.6	100	0.15	PVD		3.05±0.05
102.2.6	100	0.01	IAD	330	1.87±0.07
925.2.6	100	0.05	IAD	500	2.17±0.12
930.1.6	100	0.15	IAD		7.61±0.24
101.1.6	175	0.01	PVD		2.60±0.27
924.2.6	175	0.05	PVD	500	2.69±0.07
929.2.6	175	0.15	PVD	500	2.63±0.09
101.1.6	175	0.01	IAD		1.99±0.07
924.2.6	175	0.05	IAD	500	2.53±0.11
929.2.6	175	0.15	IAD	500	5.38±0.13
930.2.6	350	0.01	PVD	500	2.85±0.05
924.1.6	350	0.05	PVD	500	3.83±0.13
106.1.6	350	0.15	PVD	500	2.47±0.39
930.2.6	350	0.01	IAD	500	3.02±0.16
924.1.6	350	0.05	IAD	500	3.50±0.08
106.1.6	350	0.15	IAD	500	4.77±0.11
102.1.6	525	0.01	PVD	500	2.31±0.06
925.1.6	525	0.05	PVD	500	2.65±0.13
929.1.6	525	0.15	PVD	500	3.14±0.11
102.1.6	525	0.01	IAD	500	2.97±0.16
925.1.6	525	0.05	IAD	500	2.91±0.05
929.1.6	525	0.15	IAD	500	16.3±0.3
923.1.6	700	0.05	PVD	500	136±2
103.1.6	700	0.15	PVD	500	107±7
923.1.6	700	0.05	IAD	500	54.6±0.8
103.1.6	700	0.15	IAD	500	118.5±0.3

Observations of many samples indicate that roughness is not necessarily uniform; however, large variations are usually correlated with visible pits and scratches, or the boundaries between PVD and IAD regions. Remeasurements on the same samples taken about three months apart show an increase in roughness, but the relative roughness of PVD and IAD regions of the same film does not change much. Growth of oxide layers, careless handling, or some other, as yet unknown, process could be responsible for the increased rms roughness.

The roughness data on the TiN samples allows the comparison of the PVD versus the IAD regions in a relative sense without actually asserting an absolute surface roughness value for the two regions. Quoting the number recorded for each region's roughness is subject to the restriction of knowing how well anomalous pits and scratches on the surface interfered with the measurement or to what extent any of the TIS instrument's limitations affected the measurement.

7. Application 2: Chromium Coatings on Steel

Some of the first samples measured on the USNA TIS instrument were Cr coatings deposited on AISI 52100 steel. The samples are polished steel disks that were coated with Cr by an Ion-Assisted Deposition (IAD) process. Researchers at NRL developed the Cr coatings to determine their resistance to wear and corrosion.

7.1 Sample Preparation

Cr atoms from a computer-controlled e-gun source were deposited on the samples, while 40 keV Cr+ ions were simultaneously implanted into the coating and the substrate to induce greater adherence to the surface. The rate of the arriving Cr atoms was measured by a quartz crystal monitor and varied to control the arrival rate ratio of implanted ions to deposited atoms. Typical arrival rate ratios were 0.05 to 0.1, and typical coating thicknesses were 0.5 to 1 μm. The depositions were made either in high vacuum (about 0.1 mPa) or with the chamber backfilled with pure O_2 to a pressure of about 1.3 mPa.[19] Reacting with the deposited Cr, the oxygen produced coatings of Cr_2O_3.

Visually, the coatings made in high vacuum appeared frosty, suggesting roughness. The samples containing oxygen were usually shinier. Scanning electron microscope images, such as the one shown in Figure 11, verified that the coatings with oxygen were significantly smoother than those without oxygen.

Cr IMPLANTATION DURING Cr DEPOSITION

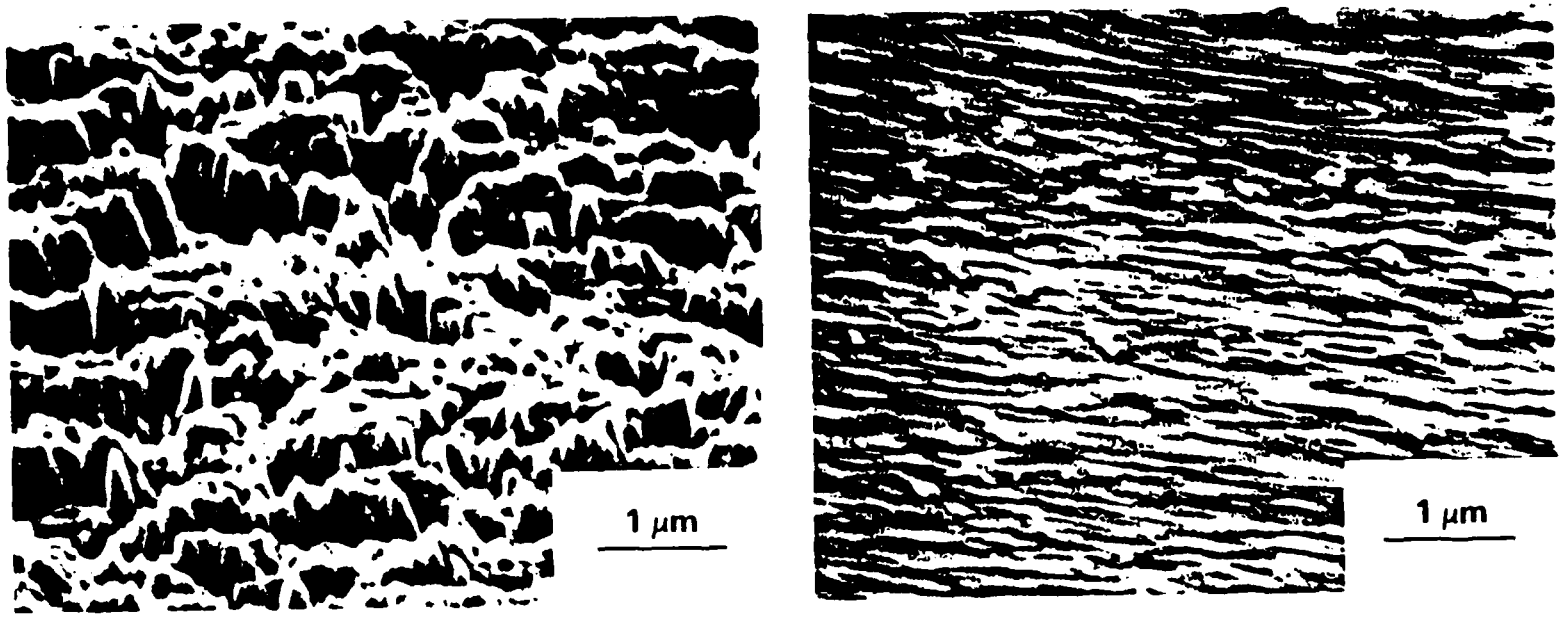

Figure 11. Scanning electron microscope (SEM) image of two Cr films produced on the surface of polished AISI-52100 steel samples using the ion-assisted deposition (IAD) technique. As discussed in the text, the film on the left was produced in high vacuum, whereas the one on the right was produced in a low-pressure oxygen atmosphere. The film on the left is clearly rougher than the one on the right. The composition of the latter film was found to be Cr_2O_3.

7.2 Results of TIS Measurements

Four coatings, two with oxygen and two without, were measured using the TIS apparatus, and the results are displayed in Table II. Consistent with the visual appearance of the sample and with SEM examinations, TIS measurements showed that coatings without oxygen are significantly rougher than those with oxygen. Samples prepared without oxygen are 2-6 times rougher in coated regions than in uncoated portions, while coatings produced in an oxygen environment are as smooth as bare steel. Even though the roughness of the oxygen-free coatings was apparent to the eye, the TIS measurements provided the first quantitative evidence that the presence of oxygen during deposition was able to almost completely suppress the roughening. These results were reported in November, 1986, at the Ninth International Conference on Applications of Accelerators in Research and Industry, at Denton, TX; a preprint of the paper submitted is in Appendix 5.

Table II. RMS roughness δ (nm) of Cr coatings on AISI 52100 steel, compared with uncoated portions of the samples.

Oxygen Backfill	δ coated	δ bare
No	25.1 ± 0.1	12.1 ± 0.2
No	74 ± 2	15 ± 2
Yes	11.1 ± 0.1	-
Yes	13 ± 1	-

8. Application 3: Ion-Beam-Mixed Mo in Al

Aluminum, one of the most popular metals for many diverse applications, owes its utility to its light weight, high strength, and relatively high corrosion resistance. However, pure aluminum is actually an intensively active metal, and its corrosion resistance results from a protective oxide layer which quickly forms on any clean aluminum surface. However, the passive layer may break down in small regions of the surface over a period of time, forming corrosion pits.[20] Researchers are not sure of the mechanisms involved in the breakdown of this passive film on aluminum, but molybdenum alloyed with aluminum has been found to circumvent the pitting and corrosion problem.

This result may at first be encouraging, but adding Mo reduces the tensile strength of bulk Al. Preliminary findings at NRL indicate that both high strength and reduced corrosion may be obtainable if a thin layer of Mo is implanted into the surface of Al. To produce a thicker and more durable layer of Mo, ion beam mixing is being investigated. In this technique, a highly energetic ion beam mixes an evaporated layer into the near-surface region of the sample producing a thick, adherent coating. The ions of the mixing beam are usually the same element as those of the deposited layer, but do not necessarily have to be.

Since ion mixing causes defects and radiation damage to the solid, atomic mobility in the material's solid structure results in migration towards these defects.[21] The ion-mixed atoms smear out into much greater depths than expected from typical ion implantation. Researchers

at NRL would like to quantify the mobility of Mo in Al. Using the relatively well-established technique of Rutherford backscattering (RBS), workers at NRL have apparently observed a diffuse Mo layer, indicating considerable mobility of Mo in Al.[20] However, a rough surface may produce a similar signal in the RBS data. An independent means of distinguishing between a rough surface and a smeared out, highly mobile layer is necessary. Because the USNA TIS instrument looks at only the rms heights of a surface, it would be an ideal technique for the purpose.

Four different types of samples were studied. A polished piece of Al had a rms roughness of 7.3±0.4 nm. This was comparable to a sample implanted with 50keV and 110keV Mo+ ions, with a rms roughness of 5.9±1.1 nm. Depositing a 28-nm-thick layer of Mo on Al from an e-gun while the substrate was at a temperature of 300°C produced a sample with a measured rms roughness of 4.4±0.4 nm. The 30-nm-thick deposited Mo-on-Al sample that had been ion-beam mixed with 10^{17} 1.8 MeV Xe+ ions/cm^2 had a rms roughness of 6.4±0.2 nm.

The roughness measured for the three types of Mo treated samples would have to vary by more than 10 nm from that of the polished Al sample to cause any concern for the interpretation of RBS data. Since that was not the case, measurements with the USNA TIS instrument support the model of a mobile Mo layer.

9. Application 4: Si_xN_{1-x} Layers

At NRL thick layers of Si_xN_{1-x} were grown on Si by ion-assisted deposition.[22] An electron gun heater evaporates Si while N was ionized and accelerated to 1000 eV. Directing the two elements at near right angles to the substrate's surface, layers of 1.5 μm and 3.0 μm thickness were produced. This technique enabled NRL to tailor the refractive index profile of the layers for use in interference filters for infrared light.

A possible unwanted side effect of depositing thick layers onto Si could be a large increase in surface roughness. Measurements with the USNA TIS instrument show a rms roughness of 2.8±0.4 nm, which is only one part in one thousand of the thickness of the deposited layer. This result is encouraging because it means that thick layers can be produced without increasing the surface roughness by a significant amount.

10. Application 5: Superlattices

Recently, researchers in the semiconductor and electronics field are attempting to develop new types of "semiconducting heterostructures" [23], called strained-layer superlattices. Thin layers of materials with different lattice constants are grown together. The crystal deforms slightly to cause a continuous transition from one layer to the next. The fact that this process can be performed means that the crystal structure can be modified to produce specific bandgap and transport properties in semiconductors.

Ion bombardment has been used to create superlattices in localized regions of the crystal structure.[23] Researchers at NRL have produced 10-nm-thick GaAs/AlAs superlattices which are in an epitaxial form. Hundreds of layers were formed, which displayed a periodic quantum well array. This superlattice has particularly high electron mobility due to a low effective electron mass.[24] TIS measurements on the rms roughness of three of the GaAs/AlAs superlattices with a thickness of 6 μm show a rms roughness of 4.1±0.2 nm. This variation in roughness is only one part in one thousand as compared to the thickness of the layer. This result demonstrates that thick superlattice layers can be produced without introducing any significant surface roughness.

11. Conclusions

A simple, easily operated, inexpensive optical instrument, based upon total integrated scatter, has been built to measure the rms surface roughness of ion-bombarded materials. The instrument was tested and refined several times in order to improve its accuracy and reliability. More improvements can be made; perhaps the most important among them would be to eliminate the low-level light scattering problem and to reduce any scraping of the specularly reflected beam at the exit hole in the hemisphere.

The TIS instrument was used to measure the rms roughness of many novel material systems. An intensive study of the rms roughness of TiN films on Si with a physical vapor deposition region and an ion-assisted deposition layer was performed. The findings indicate that ion-assisted deposition can create a smoother layer than just vapor deposition at low arrival rates and low temperatures. The physical vapor deposition layer is generally independent of the arrival rate.

Chromium coatings that had been ion-assisted deposited in a high vacuum on AISI 52100 steel were compared to Cr_2O_3 coatings produced in an oxygen atmosphere. The coatings made in vacuum were found to be rougher, whereas the oxide layer samples were about as smooth as bare steel. Implanted, ion-beam-mixed layers of Mo in Al, which may be effective for improving the corrosion resistance of Al, were found to have too little roughness to cause a misinterpretation of RBS data.

Thick Si_xN_{1-x} layers on Si varied only about one part in one

thousand in rms roughness as compared to thickness. GaAs/AlAs superlattices, currently being developed for band gap engineering, had rms roughnesses that varied less than one part in one thousand as compared to layer thickness.

The possibility exists for in situ measurements of the formation of roughness. The instrument can be modified to fit inside of a vacuum bell jar while a substrate is being coated with a substance. The coating process can be stopped at chosen times, and almost immediately the deposited substrate can be measured by the TIS instrument for surface roughness. A feedback control system logically follows.

The elegance of the TIS measuring process is that it is relatively simple, easily operated, fast, and nondestructive. Routine rms roughness measurements on many kinds of ion-bombarded materials can be the best application of the TIS instrument.

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Appendix 1: AUTO8 Program

```
]LOAD AUTO8
]LIST

 100  DIM V(2,10): DIM SP(100): DIM DI(100): DIM TIS(100): DIM RU(100)
 110  DIM A$(20): DIM S$(80): DIM DA$(10): DIM OP$(80)
 120 D$ =  CHR$ (4):G$ =  CHR$ (7):Z$ =  CHR$ (26)
 125  REM  INITIALIZE FLAGS AND RELAY ****
 126  POKE  - 16296,0:S = 1:R = 0
 150  PRINT D$;"PR#3"
 151  PRINT "USNA TOTAL INTEGRATED SCATTER APPARATUS": PRINT : PRINT
 152  PRINT "ENTER OPERATOR ID": INPUT OP$
 154  PRINT "ENTER ANALYSIS DATE": INPUT DA$
 155  PRINT "ENTER SAMPLE IDENTIFICATION STRING": INPUT S$
 156  HOME : PRINT D$;"PR#1": PRINT  CHR$ (27) +  CHR$ (49)
 157  PRINT "USNA TOTAL INTEGRATED SCATTER MEASUREMENT"
 158  PRINT "5 mW HeNe laser @ 632.8 nm": PRINT
 159  PRINT "SAMPLE IDENTIFICATION: ";S$
 160  PRINT "ANALYSIS DATE: ";DA$
 165  PRINT "OPERATOR ID: ";OP$
 170  PRINT : PRINT : PRINT
 177  PRINT "M";: POKE 36,5: PRINT "SPECULAR";: POKE 36,20: PRINT "DIFFUS
     E";: POKE 36,35: PRINT "TOTAL";: POKE 36,50: PRINT "TIS";: POKE 36,6
     5: PRINT "RMS NM";: PRINT : PRINT
 180  PRINT D$;"PR#3"
 200  REM  *****MAIN LOOP*****
 210  HOME
 220  FOR M = 1 TO 100
 225  FOR B = 1 TO 5: PRINT G$: NEXT B
 226  PRINT "USNA TOTAL INTEGRATED SCATTER APPARATUS": PRINT : PRINT
 228  PRINT "CHECK THAT THE SAMPLE IS PROPERLY POSITIONED"
 230  PRINT "TO MAKE MEASUREMENT NUMBER ";M;" ON THIS SAMPLE, ENTER R"
 232  PRINT "TO STOP BEFORE ANOTHER MEASUREMENT, ENTER S"
 234  INPUT R$
 240  IF R$ <  > "R" AND R$ <  > "S" THEN 230
 250  IF R$ = "S" THEN MM = M - 1: GOTO 800
 500  HOME : REM  ****SPECULAR MEASUREMENT****
 510  PRINT "SPECULAR INTENSITY MEASUREMENT NUMBER ";M
 515  POKE  - 16296,0
 520 S = 1
 525  FOR D = 1 TO 1000: NEXT D: REM   DELAY WHILE RELAY SWITCHES
 560  GOSUB 1000: REM   READ DVM FOR SPECULAR INTENSITY
 570  GOSUB 1200: REM  AVERAGE RESULTS
 580 SP(M) = VA
 600  HOME : REM  ****DIFFUSE MEASUREMENT****
 610  PRINT "DIFFUSE INTENSITY MEASUREMENT NUMBER ";M
 615  POKE  - 16295,0
 620 S = 2
 625  FOR D = 1 TO 1000: NEXT D: REM   DELAY WHILE RELAY SWITCHES
 660  GOSUB 1000: REM  READ DVM FOR DIFFUSE INTENSITY
 670  GOSUB 1200: REM  AVERAGE RESULTS
 680 DI(M) = VA
 700  GOSUB 1400: REM  CALCULATE TIS AND RU FOR THIS MEASUREMENT
 720  REM  ****GO BACK AND GET ANOTHER MEASUREMENT
 740  NEXT M
 800  GOSUB 1600: REM  AVERAGE RESULTS FOR THIS SAMPLE
 900  END
```

```
1000  REM  SUBROUTINE TO READ KEITHLEY
1001  FOR R = 0 TO 5
1002 V(S,R) = 0
1005  PRINT D$;"PR#4": PRINT D$;"IN#4"
1020  PRINT "RA"
1030  PRINT "LF1"
1040  PRINT "WT4";Z$;"ROT1X"
1050  PRINT "RDT";Z$;: INPUT "";A$
1060  PRINT "UT"
1070  PRINT D$;"PR#0": PRINT D$;"IN#0"
1080  PRINT D$;"PR#3"
1090 V(S,R) =  VAL ( MID$ (A$,5,11))
1092  NEXT R
1100  RETURN
1200  REM  SUBROUTINE TO CALCULATE AVERAGES
1205 VS = 0
1210  FOR R = 1 TO 5:VS = VS + V(S,R): NEXT R
1250 VA = VS / 5
1290  RETURN
1400  REM   SUBROUTINE TO CALCULATE TIS AND RU AND PRINT DATA
1410 TIS(M) = DI(M) / (SP(M) + DI(M))
1420 RU(M) = (50.387) *  SQR ( -  LOG (1 - TIS(M)))
1430 TIP =  INT (TIS(M) * 10000) / 10000
1440 RUP =  INT (RU(M) * 100) / 100
1450  PRINT D$;"PR#1"
1460  PRINT M;: POKE 36,5: PRINT SP(M);: POKE 36,20: PRINT DI(M);: POKE
    36,35: PRINT SP(M) + DI(M);: POKE 36,50: PRINT TIP;: POKE 36,65: PRINT
    RUP
1470  PRINT D$;"PR#3"
1480  RETURN
1600  REM  SUBROUTINE TO CALCULATE AVERAGE ROUGHNESS
1605 RS = 0:RA = 0:TS = 0:TA = 0:SS = 0:DS = 0
1607 R2 = 0:T2 = 0
1608  IF MM = 1 THEN 1740
1610  FOR M = 1 TO MM
1611 RS = RS + RU(M):TS = TS + TIS(M):SS = SS + SP(M):DS = DS + DI(M)
1620 R2 = R2 + RU(M) * RU(M)
1622 T2 = T2 + TIS(M) * TIS(M)
1624  NEXT M
1630 R2A = R2 / MM:T2A = T2 / MM
1635 RA = RS / MM:TA = TS / MM:SA = SS / MM:DA = DS / MM
1640 DR =  SQR ((R2A - RA * RA) / MM)
1645 DT =  SQR ((T2A - TA * TA) / MM)
1660 AR = 50.387 *  SQR ( -  LOG (1 - TA))
1662 AP = 50.387 *  SQR (TA): REM  SIMPLE APPROX TO EXPONENTIAL
1663 AP =  INT (AP * 100) / 100
1665 AR =  INT (AR * 100) / 100
1666 RA =  INT (RA * 10000) / 10000:DR =  INT (DR * 10000) / 10000
1667 TA =  INT (TA * 100000) / 100000:DT =  INT (DT * 100000) / 100000
1670  PRINT D$;"PR#1": PRINT
1672  PRINT "AVG";: POKE 36,5: PRINT SA;: POKE 36,20: PRINT DA;: POKE 36
    ,35: PRINT SA + DA;: POKE 36,50: PRINT TA;: POKE 36,65: PRINT RA
1674  PRINT : PRINT
1680  PRINT "MEAN TIS = ";TA;" +/- ";DT;" (SDOM)"
1690  PRINT "MEAN RMS NM = ";RA;" +/- ";DR;" (SDOM)"
1691  PRINT
```

```
1700  PRINT "ROUGHNESS FROM MEAN TIS = ";AR;" NM": PRINT
1710  PRINT "ROUGHNESS FROM APPROXIMATION TO EXP. = ";AP;" NM"
1720  PRINT  CHR$ (12)
1730  PRINT D$;"PR#3"
1740  RETURN
```

Appendix 2: ALIGN Program

```
]LOAD ALIGN
]

]LIST

 100 D$ =  CHR$ (4):Z$ =  CHR$ (26)
 105  POKE  - 16296,0
 110  FOR I = 1 TO 100: NEXT I
 200  PRINT D$;"PR#4": PRINT D$;"IN#4"
 210  PRINT "RA"
 220  PRINT "LF1"
 230  PRINT "WT4";Z$;"ROT1X"
 240  PRINT "RDT";Z$;: INPUT "";A$
 250  PRINT "UT"
 260 SP =  VAL ( MID$ (A$,5,11))
 300  POKE  - 16295,0
 310  FOR I = 1 TO 100: NEXT I
 400  PRINT D$;"PR#4": PRINT D$;"IN#4"
 410  PRINT "RA"
 420  PRINT "LF1"
 430  PRINT "WT4";Z$;"ROT1X"
 440  PRINT "RDT";Z$;: INPUT "";A$
 450  PRINT "UT"
 460 DI =  VAL ( MID$ (A$,5,11))
 600 TIS = DI / (SP + DI)
 610  IF TIS > 0 THEN  GOTO 630
 620 TIS = 0
 630  IF TIS < 1 THEN  GOTO 650
 640 TIS = .99999
 650 RU = (50.387) *  SQR ( -  LOG (1 - TIS))
 660  PRINT D$;"PR#0": PRINT D$;"IN#0"
 670  HOME : HTAB 15: VTAB 15: PRINT RU
 710  GOTO 105
 800  END
```

Appendix 3: Output From Typical Sample Measurement

USNA TOTAL INTEGRATED SCATTER MEASUREMENT
5 mW HeNe laser @ 632.8 nm

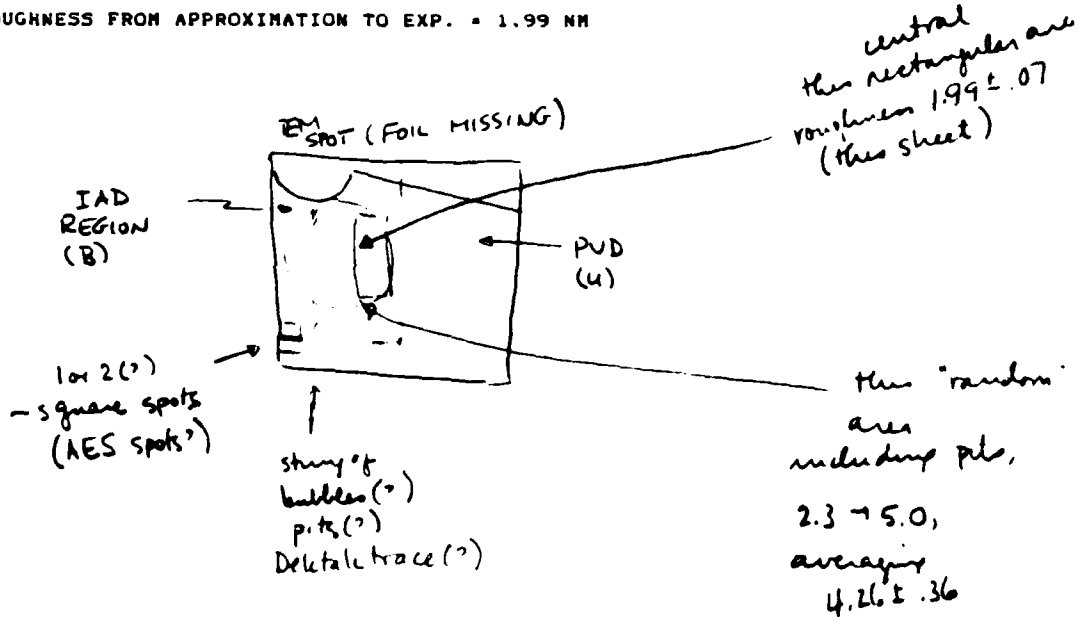
SAMPLE IDENTIFICATION: TiN 101.1.6 175 C R=0.01 BOMBARDED REGION
ANALYSIS DATE: 25 FEB 87
OPERATOR ID: CDF

M	SPECULAR	DIFFUSE	TOTAL	TIS	RMS NM
1	3.3632E-03	6.8E-06	3.37E-03	2E-03	2.26
2	3.4018E-03	5E-06	3.4068E-03	1.4E-03	1.93
3	3.3574E-03	5.4E-06	3.3628E-03	1.6E-03	2.01
4	3.339E-03	3.8E-06	3.3428E-03	1.1E-03	1.69
5	3.3166E-03	5.8E-06	3.3224E-03	1.7E-03	2.1
6	3.3722E-03	4.20000001E-06	3.3764E-03	1.2E-03	1.77
7	3.4024E-03	6E-06	3.4084E-03	1.7E-03	2.11
AVG	3.36465714E-03	5.28571429E-06	3.36994286E-03	1.56E-03	1.9876

MEAN TIS = 1.56E-03 +/- 1E-04 (SDOM)
MEAN RMS NM = 1.9876 +/- .0697 (SDOM)

ROUGHNESS FROM MEAN TIS = 1.99 NM

ROUGHNESS FROM APPROXIMATION TO EXP. = 1.99 NM



Appendix 4: March APS Abstract

Bulletin of the American Physical Society 32 (1987), p. 891

12:36

QJ 9 Optical Measurement of RMS Roughness of Ion Implanted Surfaces. C.D. FERGUSON* and F.D. CORRELL, U.S. Naval Academy, and R.A. KANT, Naval Research Laboratory.-- Ion implantation and related ion-beam processing techniques sometimes roughen the surfaces to which they are applied. Depending upon their intended use, such roughness may be either harmful or beneficial; in either case, if undetected it can cause erroneous interpretation of data gathered by most standard surface-analysis techniques. Surface profilometers and scanning electron microscopes are often tedious to use, and many lack the resolution needed to detect very fine-scale roughness that can still confuse the data interpretation. We have built and tested a simple optical instrument that measures the rms roughness, below about 100 nm, of ion-implanted surfaces. The instrument (adapted from a design developed at Polaroid Optical Engineering) actually measures the total integrated scatter (TIS) of almost normally-incident laser light, which is simply related to the rms surface roughness under certain conditions. We will describe our TIS instrument, and show results on the roughness of TiN and Cr coatings formed by ion-assisted deposition under various vacuum and thermal conditions.

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Appendix 5: Preprint of a Paper Describing TIS Apparatus

Nuclear Instruments and Methods in Physics Research B24/25 (1987) 581-583
North-Holland, Amsterdam

OPTICAL MEASUREMENT OF THE RMS ROUGHNESS OF ION-IMPLANTED SURFACES

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Ion implantation and other ion-beam processing techniques sometimes roughen the surfaces to which they are applied. If undetected, such roughness can lead to erroneous interpretation of data gathered by RBS, AES, and other surface analysis techniques. Many surface profilometers and scanning electron microscopes lack sufficient resolution to detect fine-scale roughness that can complicate the data interpretation. We have constructed a simple optical instrument to measure the root-mean-square (rms) roughness, below ~100 nm, of ion-implanted surfaces. This instrument measures the total integrated scatter (TIS) of almost-normally incident laser light, which (under certain conditions) is simply related to the rms surface roughness. This paper describes the construction and calibration of the TIS instrument, and presents preliminary results on the roughness of Cr surfaces deposited under various vacuum conditions and implanted during deposition with energetic Cr ions.

1. Introduction

Ion implantation and closely-related processes like ion-assisted deposition (IAD) and ion-beam mixing (IBM) are becoming widely used for modifying the properties of materials. Sometimes these processes also roughen or texture the surfaces to which they are applied [1]. Depending upon the material's intended use, such roughening may be either benefical or harmful; in either case, if it is undetected it can lead to erroneous interpretation of data gathered by Rutherford backscattering spectroscopy (RBS), Auger electron spectroscopy (AES) and other surface analysis techniques [2-8].

Mechanical profilometry and electron microscopy are often used to investigate surface roughness, but these techniques are relatively difficult and time consuming, and sometimes lack the resolution needed to detect very fine-scale roughness that can still affect data interpretation. A rapid, simple, reasonably quantitative method of assessing surface roughness would be useful.

One promising method, known as total integrated scattering (TIS), has been used for many years to measure the microroughness of precision optical components [9]. The TIS technique is based on the fact that a rough surface reflects light more diffusely than a smooth one does. Under conditions that may apply to many ion-bombarded surfaces, the intensity ratio of diffusely scattered light to all reflected light can be related in a straightforward way to the root-mean-square (rms) roughness of the reflecting surface. Useful rms roughness measurements can be made with very simple, inexpensive apparatus, and this technique shoudl find many applications in the field of ion-beam modification of materials.

2. Theory of total integrated scattering from slightly-rough surfaces

A surface is considered to be slightly rough if its rms roughness δ is much smaller than the wavelength λ of the light used to illuminate it. For such a surface, it is useful to define the TIS as the intensity ratio of diffusely scattered light to all reflected light. The diffuse reflectance can be calculated using scalar scattering theory, which is based on the Kirchoff diffraction integral; when the surface is only slightly rough, the TIS is given by [10]:

$$\mathrm{TIS} = 1 - \exp\left[-(4\pi\delta\cos\theta/\lambda)^2\right],$$

where θ is the angle of incidence. If the TIS from a surface is measured, then its rms roughness can be computed.

Unless great care is taken to collect light scattered at very small angles (< 0.5°) from the specular direction, the TIS technique is sensitive to only short-wavelength (< 100 μm) components of the roughness [11]. For this reason, it is sometimes difficult to compare TIS results with those of different measurement techniques, such as mechanical profilometry. Still, the speed and ease with which TIS measurements can be made suggest that this technique should be useful for routine evaluation of the roughness of ion-implanted surfaces.

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(North-Holland Physics Publishing Division)

3. Construction and calibration of the TIS instrument

The design of our TIS instrument was adapted from one developed at Polaroid Optical Engineering [12], and is shown in fig. 1. The sample is displaced ~ 1 cm from the center of an aluminized, 24-cm-diameter, acrylic hemisphere. Light of wavelength $\lambda = 632.8$ nm from a 5-mW, unpolarized, HeNe laser (Spectra-Physics model 105-2) is directed by a right-angle prism through a 0.3-cm-diameter hole in the hemisphere and onto the sample at an angle of ~ 2.5° from the normal. Specularly-reflected light exits the hemisphere through a similar hole, and is reflected from a front-surface dielectric mirror onto a 1-cm^2 silicon photovoltaic cell (Hamamatsu model S1337-1010BR). Scattered light is collected by the hemisphere and focussed onto an identical photocell positioned at a conjugate off-center point. Each photocell generates a current that is proportional to the incident light intensity, and these currents are measured using a digital multimeter (Keithley model 197/1972) interfaced to a small personal computer. The computer switches the input of the multimeter between the two photocells, collects intensity data, and calculates and averages the TIS and roughness for several points on each sample. The total cost of the instrument, except for the computer and an optical breadboard, was about $1600.

The performance of our instrument was first checked using a set of roughness standards made at the Naval Weapons Center (NWC), China Lake, for the U.S. Army [13]. This set consists of fifteen, 3.5-cm-diameter disks of fused quartz, flint glass, or BK-7 optical glass, which were mechanically polished and then aluminized. Their rms roughnesses were measured by TIS at NWC shortly after fabrication, and ranged from 0.42 to 33 nm. After the standards had been used at a number of laboratories, they were remeasured at NWC, and their roughnesses were found to have increased by as much as 80%. In fig. 2 the new NWC values (squares) and the roughnesses measured using our instrument (pentagons) are plotted versus the original roughnesses. Each point represents the average of 39 measurements for the NWC data, and about 15 measurements for ours. The two sets of remeasuments are in general agreement, except for the smoothest standards. For those, our roughness values are consistently greater than the NWC values. This could be explained by a further roughening of the standards since the last NWC measurements, but is more likely the result of low-level light scattering due to misalignment or insufficiently precise design of our instrument. This descrepancy indicates that our instrument may not be accurate for measuring roughness below ~ 2.0 nm. Additionally, experience with the similar instrument at Polaroid Optical Engineering suggests that our instrument should be accurate for measuring roughness as large as 100 nm.

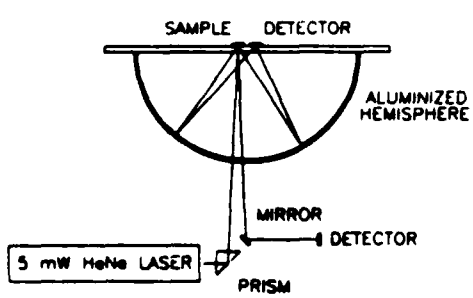

Fig. 1. Schematic diagram of the USNA TIS instrument. The detectors are connected to a relay, a digital multimeter, and a small personal computer, which are not shown.

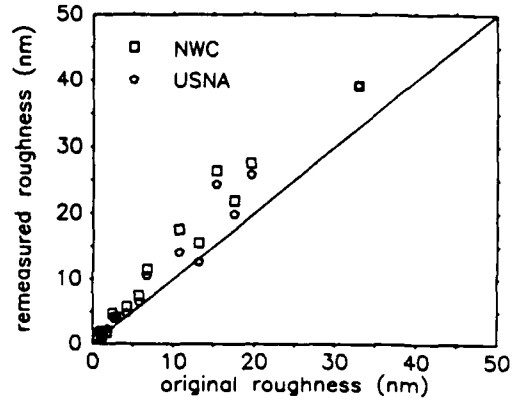

Fig. 2. Performance of the USNA TIS instrument compared with that of a state-of-the art instrument at NWC, China Lake, using a set of 15 roughness standards. Roughnesses measured using USNA and NWC instruments are plotted versus original roughnesses measaured at NWC, as discussed in the text. Diagonal line indicates equal original and remeasured values.

As a further check, the roughness of a Marz-grade Co sample polished with 9-μm alumina was measured using a Dektak profilometer and our TIS instrument. A chart-recorder trace from the profilometer was digitized, and the rms roughness of the surface was found to be approximately 27 nm. The TIS instrument yielded a value of (28 ± 3) nm. Although roughness values obtained by TIS and by mechanical profilometry are not necessarily expected to be consistent [11], this agreement is encouraging.

4. Application: ion-assisted deposition (IAD) of Cr on AISI 51200 steel

The first use of our TIS instrument has been to measure the roughness of several Cr coatings on AISI 52100 steel. These coatings were made at NRL as part

Table 1
RMS roughness (nm) of Cr coatings on AISI 52100 steel, measured using the USNA TIS instrument. R is the arrival rate ratio discussed in the text, whereas t is the estimated thickness of the Cr of Cr + O coating.

Sample	Oxygen backfill	R	t (μm)	Coated area	Bare area
A	No	0.1	-	25.1 ± 0.1	12.1 ± 0.2
B	No	0.2	0.6	74 ± 2	15 ± 2
C	Yes	-	-	11.1 ± 0.1	-
D	Yes	0.05	1.0	13 ± 1	-

of a large basic-research program whose general goal is to improve the corrosion, friction, and wear of various metals. The samples studied were polished steel disks that were Cr-coated by the IAD process [14]: while Cr atoms from a computer-controlled e-gun source were deposited on the sample, 40-keV Cr^+ ions were simultaneously implanted into the deposited coating and the substrate. A quartz-crystal monitor measured the arrival rate of the deposited Cr atoms, and allowed the computer to vary the deposition rate in order to control the arrival-rate ratio of implanted ions to deposited atoms. Typical arrival-rate ratios were 0.05–0.1, and typical coating thicknesses were 0.5–1 μm. The depositions were made either in high vacuum (about 1×10^{-4} Pa) or with the chamber backfilled with pure O_2 to a pressure of about 1.3×10^{-3} Pa. This oxygen reacted with the deposited Cr to produce coatings with the composition (measured by RBS and AES) Cr_2O_3. The density of the coatings is still unknown.

Some samples coated in high vacuum appeared milky or frosty, suggesting roughness; whereas those containing oxygen usually appeared shinier, or smoother. Scanning electron microscope observations supported these conclusions. Four coatings, two with oxygen and two without, were measured using the TIS instrument, and the results are presented in table 1. These results may be summarized by noting that the coatings without oxygen are 2–6 times rougher than the bare or uncoated parts of the samples, whereas the coatings containing oxygen are about as smooth as the bare parts. While the roughness of the oxygen-free coatings was apparent even to the eye, the TIS measurements provided the first quantitative evidence that the presence of oxygen during deposition was able to almost completely suppress the roughening.

5. Conclusions

We have constructed a very simple, inexpensive TIS instrument, and have shown that it can be used to obtain rapid, reasonably quantitative measurements of the roughness of ion bombarded surfaces. We believe that there should be many useful applications in the field of ion-beam modification of materials.

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